MY VISION OF THE ORGANIC ENVIRONMENTAL LABORATORY IN THE 21ST CENTURY

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Introduction

I want to thank my editor for the opportunity to express my own personal vision in a brief overview of how I think that the organic environmental analytical laboratory will operate in the first decade of the 21st Century. As we approach the millennium, we find that many environmental laboratories are still using some of the same basic sample preparation techniques and determinative methods that they have used since the 1980s. Many of these sample preparation methods are either lengthy and solvent-intensive, e.g., Soxhlet extraction and continuous liquid-liquid extraction, or labor-intensive and solvent-intensive, e.g. ultrasonic extraction and separatory funnel shake extraction. A few can even be downright dangerous, such as the sample preparative methods for phenoxyacid herbicides by gas chromatography (GC) using ether as the extraction solvent and diazomethane for derivatization as methyl esters. Gas chromatography/mass spectrometry (GC/MS) and GC with selective detectors have been the determinative methods of choice. These "commonly-used" methods usually generate data of sufficient quality to meet the data quality requirements for most of the current environmental applications. However, with the dawn of the new century we should be thinking about how we can do it better and more cost effectively.

New requirements to collect extraction solvents for proper disposal as hazardous wastes, rather than to allow them to boil off up through the hood exhaust have significantly increased the cost of doing business in the traditional way. In addition, the use of some of the traditional sample handling procedures for volatile analytes in soil and other solid matrices have resulted in the underestimation of actual volatiles content by orders of magnitude. Finally, the traditional approach to sampling and analysis has primarily been to collect the samples in the field and ship them off to a fixed laboratory for analysis.

I believe that with the coming of the millennium we will see, and as analytical chemists we will implement, a paradigm change in the way that we approach organic analyses. Several factors will influence this change, the most important of which I will address in a little detail in this article. These major factors include: 1) the utilization of the performance based measurement system (PBMS) approach for environmental monitoring; 2) the use of "green" methods; 3) the improved use of screening methodology; 4) the use of more on-site analysis; 5) the use of improved sampling and sample handling techniques, and 6) the increased use of high performance liquid chromatography (HPLC) in environmental analysis.

Performance Based Measurement Systems (PBMS)

Contrary to popular belief in many sectors of the environmental community, PBMS is not a "new" approach to analytical chemistry. It is as old as science, since it is an iteration of the scientific method. Simply put, the PBMS approach requires an analyst to be able to address three basic scientific questions before initiating any analytical or monitoring activity. These questions are as follows:

1) What is the purpose of this project, or why am I doing this?
2) How will the data be used in the decision-making process?
3) What quality of data is needed to support the decision, or how good does the data have to be?

In the world of prescriptive methodology that guides much of environmental analysis today, maybe this is a new concept. However, using the PBMS approach is the way that chemists work in just about any other industry and chemical discipline.

The key paradigm shift in using the PBMS approach is that the focus is on demonstrating that the data quality criteria for a particular application have been met, rather than whether a particular published method was followed. This will also require the same paradigm shift for enforcement, which will need to focus compliance audits on whether the data generated met the regulatory data quality requirements, rather than focusing these audits on laboratory quality systems. PBMS allows for much greater flexibility and cost effectiveness in methods selection, since it allows the analyst to focus on generating analytical and QC data to meet specific project needs, rather than trying to globally address all of the target analytes in a published method, as is done in many cases today. However, once the method selection is made and an SOP generated, it must be followed, unless there is a compelling reason not to do so which must be documented.

PBMS is not a *laissez faire* approach. In order for it to work to its fullest potential, regulators will have the responsibility of including the data quality criteria that need to be met directly in the regulations and permits, and not just quote some published method number. It will require generation of better documentation and record keeping on the part of the generators and the laboratory than we do now. Contrary to popular belief, there will not be a rush to generate brand new methods for environmental applications under PBMS. Most of the analytical flexibility will involve the specific tailoring of existing methods to address specific project needs. The minimum documentation necessary to support these analytical activities should include a sampling and analysis plan (SAP) which includes, at a minimum: 1) sampling plan delineating the number and types of samples needed; 2) detailed SOPs of all of the analytical methods to be used (not just published method numbers); 3) QC procedures that need to be performed in order to demonstrate that the data generated meets project requirements; and 4) proficiency demonstrations that show that the analyst(s) can perform the method, and that the method demonstrates appropriate performance to meet the data quality needs of its intended application. I expect that in the first decade of the new century that there will be a sufficient number and variety of performance evaluation (PE) samples in a variety of matrices that will greatly facilitate the

laboratories' ability to do these performance demonstrations.

In summary, I believe that laboratories will adopt the PBMS approach for their operations. PBMS will allow the laboratories to operate more cost effectively by being much more selective in their choice of methodology and minimizing requirements to do extraneous analyses and unnecessary QC procedures. Also, I expect that the overall quality of environmental data should improve because laboratories will be required to demonstrate and document that the methods that they select actually work for the applications for which they are intended. Finally, the experience of our enforcement people has shown that any data that were generated under conditions that were scientifically defensible were also legally defensible.

"Green" Methods

"Green" methods have become a major buzzword in several EPA Programs over the past several years. Some are farther along than others in their adoption of these methods. In the RCRA Program, for over ten years we have tried to minimize the use of hazardous substances in our analytical methods. (On my last industrial job, in the fats and oils industry, my Control Laboratory generated more RCRA hazardous waste from our analytical processes, i.e., solvents, than was generated from the production facility.) We have published several extraction methods for organic compounds which cut down significantly (90-95% reduction) on solvent use, beginning with the Automated Soxhlet method (Method 3541) in Update II of SW-846 back in 1995, and continuing with Update III in 1997. Several more have been published in draft form and can be found on the OSW Methods Team Homepage on the Internet.

My vision of the organic extraction laboratory of the early part of the next century is one where we no longer see any large banks of Soxhlet extractors or continuous liquid-liquid extractors using copious quantities of solvent which need to be collected either for disposal or redistillation. (This is like modern-day record stores, which no longer stock records, only cds and cassettes). I also expect to see the demise of the labor- and solvent-intensive ultrasonic extraction and separatory funnel extraction methods as well for the same reasons.

I would expect to see "green" methods, such as pressurized fluid extraction (PFE) or microwave extraction used as the standard methods for the extraction of solid matrices, and I would expect to see solid phase extraction (SPE) used as the standard method for the extraction of aqueous matrices. These methods have extraction efficiencies comparable to or better than the most rigorous of the older methods, can be automated, can be performed in minutes instead of hours, are not labor intensive, and use between 10 and 60 mLs of solvent instead of the 500 to 600 mL of solvent as do Soxhlet and continuous liquid-liquid extraction.

Other "green" methods which are rapid and use either very little or no hazardous solvents, like supercritical fluid extraction (SFE) and some microextraction methods, will also have a place in the environmental laboratory of the 21st Century. However, I expect that they will have limited use for

some very specific applications, e.g., extraction of a specific class of analytes from a complex sample. I do not expect that they will have the widespread marketability of either PFE or SPE.

Screening Methods

With the advent of a solid PBMS program, the effective use of appropriate qualitative and quantitative screening methods should substantially increase. They can be used either directly in the field or in fixed laboratories, in many cases to directly answer analytical questions. OSW has pioneered the use of new screening technologies by incorporating several of them into SW-846. These technologies include immunoassays and colorimetric kits using a variety of chemical reactions, and are applicable to the analysis of different classes of organic compounds, e.g., PAHs, PCBs, explosives, organochlorine compounds, pesticides and herbicides to name a few.

Quantitative screening methods, i.e., methods that determine whether the target analyte(s) are present at a particular action level at a designed-in confidence limit, can be used either in the field or in the laboratory. Field applications include mapping of contaminated hot spots for a hazardous waste site characterization to determine whether a specified site cleanup level has been reached. With the proper use of these methods, only a small percentage, i.e., a spot check, of analyses need to be run for confirmation analysis using conventional determinative methods. Laboratory uses will be screening samples to make sure that they do not contain concentrations of target analytes that will overwhelm instrument and detector capabilities and thus prevent significant instrument downtime. Effective use of screening methods can improve the overall quality of data generated for environmental projects because the lower cost per analysis allows many more samples to be analyzed than would be the case using conventional laboratory methods.

I also expect to see some instrumental screening methods, e.g., direct-sampling ion trap mass spectrometry and ion mobility spectrometry to be used for site characterization applications. Other instrumental techniques which I expect to achieve significant use and applicability in environmental projects include on-site sensors, e.g., immunosensors, fiber optics, etc., and continuous emission monitors (CEMs) for monitoring groundwater and other down-hole monitoring applications.

On-Site Analysis

Another paradigm shift that I expect to see (in many cases it is already happening) is the move away from sending samples off to a fixed lab for analysis to performing these analyses on-site. This will lead to the increased use of mobile laboratories and screening methods directly in the field. Most modern analytical instrumentation can be adapted for use in a mobile laboratory (I have even seen a case where a triple-quad HPLC/MS system was mounted in a van as a mobile lab) or even directly in the field, e.g, on the back of a pickup truck.

Once again, using direct on-site analysis and the PBMS paradigm for project planning, we can

expect to see significant improvement in the quality of analytical data that is generated. Data for the analysis of volatile organics would be a case in point. Even with the significant improvements that we have made in the past few years in sample handling procedures, there are still some analyte losses due to transport and transfers. The ability to collect a sample for volatile organics, and run it immediately through the instrumental system with minimal sample handling and no shipping off-site, would appear to be the best way of optimizing volatiles data.

Rugged field-portable instruments will be a big focus in the next century. GC and GC/MS instruments that have sufficient sensitivity to meet project-specific data quality objectives (DQOs) will have a widespread use. Once again, the ability to perform rapid, on-site analysis in support of remediation projects can significantly reduce costs for these operations. This need will also have the potential to expedite the use of some of the new fast GC columns that some of the major manufacturers are now marketing. Some of these columns can do a BNA scan in about 6 minutes with resolution roughly comparable to standard 35- to 40-minute capillary runs.

Use of Improved Sampling and Sample Handling Techniques

One of the ways to improve the quality and increase the quantity of environmental data is to improve how sample strategies are designed and how samples are collected. In these days of diminishing resources, and I do not expect that things will improve much in this area in the foreseeable future, proper sampling is a key to generating the appropriate data needed to make informed environmental decisions. The PBMS paradigm includes sampling as a key element to do this as a significant component of the SAP. Due to the heterogeneous nature of most hazardous waste matrices, it is not feasible to get a "representative sample". However, with the use of proper sampling strategy, one can obtain samples that are "representative of the waste". OSW will publish a significantly revised Chapter Nine of SW-846 which includes substantial guidance on sampling strategy and sampling methods later this year. We expect that it, along with a new series of ASTM sampling standards, will become the basis for addressing hazardous waste sampling issues at the turn of the millennium.

The issue of sample integrity, particularly for solid samples to be analyzed for volatile organics, is one that has been extensively addressed through the 1990s. The idea of sampling solid matrices for volatiles and preventing the loss of analytes prior to analysis by preserving sample integrity has finally gained acceptance. Several sampling devices and preservation techniques are currently available and in use that protect sample integrity between the time of collecting the sample in the field and analyzing it in either the fixed or mobile laboratory. Use of these devices and techniques has already resulted in a significant improvement in the quality of volatiles data (by orders of magnitude) being generated. I expect to see continued refinements to improve these sampling and preservation techniques over the course of the next few years.

High Performance Liquid Chromatography (HPLC)

HPLC is one of the most versatile analytical techniques available, but is grossly underutilized in organic environmental laboratories. Some major industries, particularly the pharmaceutical industry, use it as their primary analytical tool for organic analysis. Some environmental laboratories use a variant for inorganic analyses, i.e., ion chromatography, but very few actively use HPLC for organic analysis..

The two major applications for HPLC in organic laboratories today are the analysis of explosives and the analysis of formaldehyde and other carbonyl compounds. Lesser applications include the analysis of N-methyl carbamates (with post-column derivatization) and some PAHs. The generation of solvents which must be handled as hazardous waste with the old-style wide-bore columns may be a deterrent to current use.

However, I believe that with the use of new capillary-type HPLC columns which significantly reduce solvent waste, as well as the availability of several new methods and the implementation of lower regulatory levels for some analytes based on risk analyses, that the use of HPLC methodology will significantly increase over the next decade. In addition to the current uses for explosives and formaldehyde, I would expect to see additional applications including 1) determination of phenoxyacid herbicides, replacing the dangerous extraction and derivatization steps; 2) determination of carbamates for LDR compliance using HPLC/MS (HPLC/MS is the only technique currently available that can do all 28 additional analytes in a single method); 3) increased analysis of PAHs as health-based regulatory limits decrease beyond the normal range of GC/MS; 4) analysis of phenols along with the phenoxyacid herbicides; and 5) analysis of pesticides, particularly for thermally labile compounds.

Summary

In summary, while I may tend to be optimistic,I predict that we will go back to being scientists in the next century and that we will adopt the PBMS paradigm, which will lead us to more cost effective analyses and better quality data upon which to base environmental decisions. We will significantly reduce the quantities of hazardous waste generated by analytical laboratories by incorporating the use of “green” methods. We will see an increase in the use of quantitative screening methods for making environmental decisions. We will see the focus of laboratory analysis shift from fixed laboratories to on-site facilities. We will better appreciate the significance of proper planning for types and number of samples, and data generated will be more “representative of the waste” being characterized. Finally, we will see the emergence of the use of some “non-conventional” methodologies including techniques like HPLC(/MS) and immunosensors.